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54: A hair cosmetic composition

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Specification

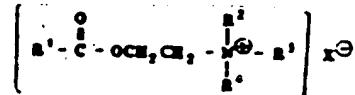
1. Title of invention

A hair cosmetic composition (hair treating composition)

2. Claims

1) ~~A hair cosmetic composition~~ containing 0.1 - 3.0 weight % of quaternary ammonium salt having the following general formula (1) and 0.1 - 10 weight % of amino acids, wherein the weight ratio of the said quaternary ammonium salt / amino acids is 1/9 - 9/1;

the general formula;



(In the formula, R1 is C11 - C23 alkyl group; R2, R3 and R4 are C1 - 3 alkyl group or hydroxy alkyl group; X is halogen atom or alkyl sulfuric acid having Cl or C2 alkyl group.).

2) The hair cosmetic composition described in the claim (1), wherein the amino acids are one or more than two kinds of amino acids selected from glycine, L-valine, L-isoleucine, L-serine, L-threonine, L-phenylalanine, L-tryptophane, L-proline, L-aspartic acid, L-glutamic acid, L-histidine, L-arginine, L-lysine and L-histidine.

3. Detailed explanation of the invention

The present invention refers to a hair cosmetic composition showing an excellent combing effect which is superior to that of the conventional cationic surfactants.

In general, a hair is covered with animal and vegetable oils such as hair cream, hair oil, etc.; and sebum secreted from the hair follicle. When the hair is washed with a soap and synthetic detergent, the oil/fat and/or sebum are excessively removed. As a result, the hair loses its smoothness and becomes hard and rough. In this case, the

combing of hair becomes rough and broken and branched hairs are generated.

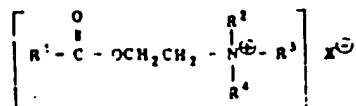
In order to solve the said problems, various kinds of hair cosmetic compositions which can treat the hair after washing have been developed and utilized. These hair treating compositions contain quaternary ammonium salts such as dialkyl dimethyl ammonium chloride, alkyl trimethyl ammonium chloride, etc. as the major active ingredient.

Some of the said quaternary ammonium salts are hardly soluble in water and some are easily soluble in water. Consequently, the hair treating compositions may be " rinse type " or " non-rinse type ".

In order to prevent squeaking and tangling of hairs after washing and to provide a fine finishing of hairs after washing, the said quaternary ammonium salt must be adsorbed on the hairs.

On the other hand, a recent trend of manufacturing the cosmetic compositions is to use safe ingredients selected from natural substances. Under the said circumstance, the present inventors have conducted many studies and found that the safety of specific choline derivatives is excellent. Moreover, the said choline derivatives have fine surface activity and demonstrate a high adsorption on the hair. Thus, the present invention was accomplished by utilizing the said knowledges. The hair cosmetic (treating) composition described in the present invention can be used in both " rinse type " and " non-rinse type " and is able to generate an excellent hair softening activity.

The purpose of the present invention is to provide a hair cosmetic (treating) composition capable of demonstrating excellent effects on the hair which can generate a soft hair. The said purpose is accomplished by a hair cosmetic composition containing 0.1 - 3.0 weight % of quaternary ammonium salt having the following general formula (I) and 0.1 - 10 weight % of amino acids, wherein the composition ratio of the said quaternary ammonium salt / the said amino acid (weight base) is 1/9 - 9/1.



(In the formula, R1 is C11 - 23 alkyl group; R2, R3 and R4 are C1 - 3 alkyl group or hydroxy alkyl group; X is halogen atom or alkyl sulfuric acid group having C1 or C2 alkyl group.).

Quaternary ammonium salts having the said general formula (I) may be called acylated choline compounds. The concentration of the said acylated choline in the cosmetic composition described in the present invention is 0.1 - 3.0 weight %, preferably 0.1 - 2.0 weight %.

If the concentration of the quaternary ammonium salt is less than 0.1

weight %, the softening effect can not be achieved. If the concentration of the quaternary ammonium salt is higher than 3.0 weight %, the treated hair becomes sticky and heavy and moreover, the softening effect of the composition is reduced.

It is very important in the present invention that R1 of the said general formula (I) is C11 - 23 alkyl group. If R1 is C11 or smaller, the water solubility increases and the adsorption on the hair is reduced, and the safety is also reduced. If R1 exceeds C23, the oil-solubility increases and the adsorption on the hair is reduced. Consequently, a preferable R1 is C15(? illegible) - C21 alkyl group.

Preferable practical examples of acylated choline compounds in the present invention are shown in the following.

choline stearyl ester halide (R1 = C17 alkyl)

choline palmityl ester halide (R1 = C15 alkyl)

choline myristyl ester halide (R1 = C13 alkyl)

choline lauryl ester halide (R1 = C11 alkyl)

choline tallowyl ester halide (R1 = C??(illegible) - 17 alkyl)

choline stearyl ester sulfate (R1 = C17 alkyl)

choline eicosyl ester halide (R1 = C19 alkyl)

choline docosanyl ester halide (R1 = C23 alkyl)

choline tetracosanyl ester halide (R1 = C?? alkyl)

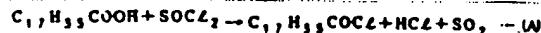
choline eicosyl ester sulfate (R1 = C19 alkyl)

choline docosanyl ester sulfate (R1 = C12 alkyl)

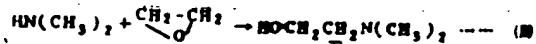
A method of synthesizing choline stearyl ester halide (stearoyl choline chloride) which is one example of acylated choline compounds described in the present invention is explained in the following.

A laboratory scale synthetic method:

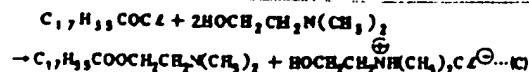
Stearic chloride (C17H35COCl) is obtained by allowing stearic acid prepared by hydrolysis of hydrogenated beef tallow to react with thionyl chloride (Reaction A).



Dimethylamine ($\text{HOCH}_2\text{CH}_2\text{N}(\text{CH}_3)_2$) is prepared by adding ethylene oxide to dimethyl amine (Reaction B).

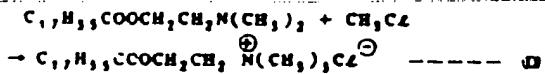


Two folds (mol base) of dimethyl ethanolamine (DME) is allowed to react with the said stearic chloride under no solvents nor catalysts, and stearoyl dimethyl ethanolamine ($C_{17}H_{35}COOCH_2CH_2\text{N}(\text{CH}_3)_2$) is obtained (Reaction C).



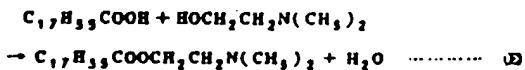
After extracting the said stearoyl dimethyl ethanolamine with ether,

the final product stearoyl choline chloride ($C_{17}H_{35}COOCH_2CH_2N(CH_3)_3$ -Cl⁻) is obtained (Reaction D).

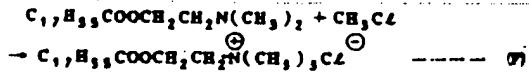


Industrial scale synthetic method:

Stearic acid is heated at high temperature (approximately 180° C) and after melting, DME is added dropwisely. After completing the reaction, water is removed under reduced pressure, and stearoyl dimethyl ethanolamine ($C_{17}H_{35}COOCH_2CH_2N(CH_3)_2$) is obtained (Reaction E).



The final product stearoyl choline chloride is obtained by quaternarizing stearoyl dimethyl ethanolamine with methylene chloride (Reaction F).



The second essential ingredient of the composition described in the present invention is amino acids, and the concentration of the said amino acid in the hair treatment composition is 0.1 - 10.0 weight %, preferably 0.1 - 5.0 weight %.

If the concentration of the amino acid is less than 0.1 weight %, the purpose of the present invention can not be achieved. On the other hand, if the concentration of the amino acid exceeds 10.0 weight %, the hair becomes sticky and heavy.

Typical examples of the said amino acids used in the present invention are glycine, L-valine, L-isoleucine, L-serine, L-threonine, L-phenylalanine, L-triptophane, L-cysteic acid, L-proline, L-aspartic acid, L-glutamic acid, L-histidine, L-arginine, and L-lysine. These amino acids may be used singly or a combination of more than two compounds. Among these amino acids, preferable ones are glycine, L-isoleucine, L-proline, L-histidine, L-arginine, L-lysine and L-cysteic acid.

One of the most important elements of the present invention is that the composition ratio of quaternary ammonium salt having the said general formula (I) and amino acid (weight base; (I) / amino acid) is 1/9 - 9/1, preferably 3/7 - 7/3. If the composition ratio of (I)/amino acid is less than 1/9, the purpose of the present invention can not be achieved. On the other hand, if the composition ratio exceeds 9/1, the

combing becomes rough.

In the hair cosmetic (treating) composition described in the present invention, other conventional ingredients may be added as long as the said ingredients do not interfere the effects of the present invention. These ingredients are listed in the following.

The said ingredients are mono-alkyl type or dialkyl type quaternary ammonium salts such as stearyl trimethyl ammonium chloride, hydrogenated beef tallow alkyl trimethyl ammonium chloride, eicosyl trimethyl ammonium chloride, heneicosyl trimethyl ammonium chloride, etc.; hydrocarbons such as liquid paraffin, vaseline, solid paraffin, squalane and olefinoligomers, etc.; esters such as isopropyl myristate, isopropyl palmitate, stearyl stearate, octyldodecyl myristate, octyl dodecyl oleate, 2-ethylhexanic acid triglyceride, etc.; higher alcohols such as cetanol, stearylalcohol, cetostearylalcohol, behenylalcohol, hexyl decanol, etc.; emulsifying agents such as glyceryl monostearate, sorbitane monopalmitate, polyoxyethylene cetylether, polyoxyethylene stearic acid ester, polyoxyethylene sorbitane monolaurate, etc.; cellulose derivatives such as methyl cellulose, hydroxyethylcellulose, hydroxypropylcellulose, cationized cellulose, etc.; viscosity regulating agents such as natural polymers, etc.; moisturizers such as ethyleneglycol, propyleneglycol, 1,3-butyreneglycol, glycerine, sorbitol, etc.; other solvents; aseptic agents; perfumes and other minor ingredients.

The hair cosmetic (treatment) composition described in the present invention can be used in various kinds of hair care and treating products such as hair rinse, spraying or dispenser type hair conditioner, hair lotion, etc.

The effects of the present invention are explained in detail in the following practical examples and comparative examples. Before exhibiting the practical examples and the comparative examples, a test method used in the said examples is explained in the following.

Test method:

A 0.5 g of test sample is directly applied on a bundle of hairs (5 gr, 20 cm) treated with a shampoo and the treated hair sample was dried at 25° C under 65 % RH. After standing overnight, the softness of the hair is evaluated by a sensory test conducted by 20 examiners. The standard sample is the hair treated with a control composition consisting of 1.0 weight % of stearyl trimethyl ammonium chloride, 3.0 weight % of cetostearyl alcohol (a mixture of one part of cetylalcohol and one part of stearylalcohol), 5.0 weight % of propyleneglycol, and water (as a balance). The hair sample treated with the test hair cosmetic composition is compared with the hair sample treated with the said control composition. The evaluation is based on 5 point score system.

Evaluation standard

0 : excellent (superior)

O : better (slightly superior)

Δ : same as the control/standard

x : slightly inferior

xx : inferior

The concentrations of the ingredients used in the following examples are based on " weight % ".

Experiment 1:

Four different kinds of hair cosmetic compositions consisting of the ingredients listed in the following Table 1 were prepared and the performance of these compositions were evaluated. The results are shown in Table 1.

Table 1:

| | a 対馬内 | b 比較 | 1 | 2 | 3 |
|---|---------------------------|------|----|-----|----|
| c | アテナリウムアリウム アンテナリウムアリウム | 10 | 1 | 1 | 1 |
| d | アシル化コリンアミン | | 10 | 0.5 | |
| e | アミノ酸アミン | | | 0.5 | 10 |
| f | アロピレンアリコール | 50 | 50 | 50 | 50 |
| g | ■ ■ ■ | — | h | — | — |
| i | アシル化コリン/アミノ酸 | | | 1/1 | |
| j | ■ ■ ■ ■ ■ | △ | △ | ○ | × |

a: practical examples, b: comparative examples, c: stearyl trimethyl ammonium chloride, d: acylated choline A *1, e: amino acid A *2, f: propyleneglycol, g: purified water, h: balance, i: acylated choline / amino acid, j: combing condition.

*1: choline stearyl ester chloride (R1 is Cl7 alkyl; R2, R3 and R4 are methyl; X is Cl.).

*2: L-proline

According to the results shown in Table 1, it is obvious that a combination of acylated choline and amino acid demonstrated excellent effect.

Experiment 2:

Eleven different kinds of hair cosmetic compositions containing various composition ratios of acylated choline and amino acid were prepared, and the performance of the said compositions were evaluated. The results are shown in Table 2.

Table 2:

a: practical examples, b: comparative examples, c: acylated choline A *1, d: amino acid A *2, e: propyleneglycol, f: acylated choline/amino acid ratio, g: combing condition.

*1 and *2 are same as described in the experiment 1.

As shown in Table 2, it is evident that a fine combing condition was achieved when the composition ratio of acylated choline / amino acid was 1/9 - 9/1.

Experiment 3:

The following hair cosmetic compositions containing various kinds of acylated choline compounds were prepared and the performance of the said compositions were evaluated. The results are shown in Table 3.

Table 3:

| | | | | | | | | |
|---|----------------|-----|----|---------|----|----|----|----|
| | a | 実施回 | 10 | 11 | 12 | 13 | 14 | 15 |
| | b | 実施回 | 7 | | | | 8 | |
| c | 実施回 | 10 | | | | | 15 | |
| | b ² | 10 | | | | | | |
| | c ² | 10 | | | | | | |
| | d ² | 10 | | | | | | 15 |
| | e ² | 10 | | | | | 15 | 15 |
| | f ² | 10 | | | | | | |
| d | 実施回 | 10 | 10 | 10 | 10 | 10 | 10 | 15 |
| e | 実施回 | 10 | 10 | 10 | 10 | 10 | 10 | 15 |
| f | 実施回 | 10 | 10 | 10 | 10 | 10 | 10 | 15 |
| g | 実施回 | 10 | 10 | 10 | 10 | 10 | 10 | 15 |
| h | * | a | b | balance | | | | |
| | 実施回 | x | o | o | o | o | o | o |

a: practical examples, b: comparative examples, c: acylated choline compounds A *1, B *2, C *3, D *4, E *5, F *6, G *7, d: amino acid B

*8, e: acylate choline / amino acid ratio, f: propyleneglycol, g: water, h: combing condition.

- *1 : choline stearyl ester chloride (R1 = C17 alkyl)
- *2 : choline capryl ester halide (R1 = C7 alkyl)
- *3 : choline lauryl ester halide (R1 = C11 alkyl)
- *4 : choline myristyl ester halide (R1 = C12 alkyl)
- *5 : choline palmityl ester halide (R1 = C16?? alkyl) (C18??)
- *6 : choline eicosyl ester halide (R1 = C18?? alkyl) (C19??)
- *7 : choline hexaconvyl ester halide (R1 = C26?? alkyl)

(Wherein R2, R3 and R4 of *1 to *7 are methyl; X is Cl.).

As shown in Table 3, it is evident that the performance of the hair cosmetic compositions containing acylated choline compounds having non-specific alkyl group (R1 was not in the range of C11 - C23) prepared in the comparative examples 7 and 8 were unsatisfactory.

Experiment 4:

Ten different kinds of hair cosmetic compositions containing various kinds of amino acids and ingredients listed in Table 4 were prepared, and the performance of the compositions were tested. As shown in Table 4, the performance of the compositions prepared in the practical examples 16 - 25 demonstrated excellent combing effect.

Table 4:

| | a | b | c | d | e | f | g | 16 | 17 | 18 | 19 | 20 | 21 | 22 | 23 | 24 | 25 |
|---|---|---|---|---|---|---|---|----|----|----|----|----|----|----|----|----|---------|
| b | | | | | | | | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 |
| c | | | | | | | | 10 | | | | | | | | | |
| | | | | | | | | | 10 | | | | | | | | |
| | | | | | | | | | | 10 | | | | | | | |
| | | | | | | | | | | | 10 | | | | | | |
| | | | | | | | | | | | | 10 | | | | | |
| | | | | | | | | | | | | | 10 | | | | |
| | | | | | | | | | | | | | | 10 | | | |
| | | | | | | | | | | | | | | | 10 | | |
| | | | | | | | | | | | | | | | | 10 | |
| | | | | | | | | | | | | | | | | | 10 |
| d | | | | | | | | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 |
| e | | | | | | | | | | | | | | | | | balance |
| f | | | | | | | | | | | | | | | | | |
| g | | | | | | | | | | | | | | | | | |

a: practical examples, b: acylated choline A *1, c: amino acid C *9, D *10, E *11, F *12, G *13, H *14, I *15, J *16, K *17, L *18, d: propyleneglycol, e: water, f: acylated choline / amino acid, g: combing condition.

*9 : glycine

*10: L-cysteic acid

*11: L-serine/L-valine (weight ratio = 1/1)

- *12: L-threonine
- *13: L-phenylalanine
- *14: L-tryptophane/L-glutamic acid (weight ratio = 1/1)
- *15: L-aspartic acid
- *16: L-histidine
- *17: L-arginine
- *18: L-lysine

Experiment 5:

Two kinds of hair rinse compositions consisting of the ingredients listed in the following Table 5 were prepared and the performance was evaluated. The method of testing the performance of the hair rinse composition was carried out as follows; in other words. 0.5 g of the test hair rinse composition was applied directly on a bundle of hairs (5 g, 20 cm) treated with shampoo (the temperature of the hair rinse composition was 40°C) and the composition was spread uniformly with hands. After rinsing followed by drying, the smoothness of the hair was evaluated.

Table 5:

| a | b | c | |
|---------------------------------------|----|-----|--------------|
| | | 比較例 | 実験例 |
| d アシル化コリン *1 | | 1.0 | 1.0 |
| e アミノ酸 *2 | | - | 1.0 |
| f セタノール | | 1.5 | 1.5 |
| g ポリオキシエチレン硬化ヒマシ油トライグリセリド (EO:10 mol) | 1 | 1 | |
| h プロピレングリコール | 5 | 5 | |
| i * 香料 | ■■ | ■■ | small amount |
| j e 色素 | • | • | small amount |
| k ■ ■ * | ■■ | ■■ | balance |
| l ■ ■ , ■ | ○ | ○ | |

a: composition ingredients, b: comparative example, c: practical example, d: acylated choline A *1, e: amino acid A *2, f: cetanol, g: polyoxyethylene hardened castor oil triisostearate (EO:10 mol), h: propyleneglycol, i: perfume, j: coloring agent, k: purified water, l: combing condition.

As shown in Table 5, an excellent effect was obtained when acylated choline and amino acid were combined.